

Effect of Acidulated Phosphate Fluoride and Casein Phosphopeptide–Amorphous Calcium Phosphate Application on Shear Bond Strength of Orthodontic Brackets

Defne Keçik^a; Sevi Burçak Çehreli^b; Çağla Şar^c; Bahtiyar Ünver^d

ABSTRACT

Objective: To evaluate the effect of a recently introduced prophylactic agent, casein phosphopeptide–amorphous calcium phosphate (CPP-ACP), on shear bond strength of brackets and compare it with the effect of acidulated phosphate fluoride (APF).

Materials and Methods: Forty-eight freshly extracted mandibular bovine incisors were used. Teeth were randomly divided into four groups ($n = 12$) as follows: group 1 served as control, and no pretreatment was performed on the enamel; group 2, enamel was treated with 1.23% APF and CPP-ACP, respectively; group 3, enamel was treated with CPP-ACP; and group 4, enamel was treated with 1.23% APF for 4 minutes. In all groups, brackets were bonded using a conventional acid-etch and bond system (Transbond XT, 3M Unitek, Monrovia, Calif). Bonded specimens were first stored in deionized water at 37°C for 24 hours, subjected to thermal cycling for 1000 cycles, and further stored in distilled water for 6 weeks before debonding procedures. After debonding, teeth and brackets were examined under a stereomicroscope at 10× magnification for any adhesive remaining, in accordance with the modified adhesive remnant index.

Results: The shear bond strengths of all experimental groups were significantly higher than that of the control group ($P < .01$). There was no significant difference between the shear bond strengths of the experimental groups ($P > .05$).

Conclusion: The use of CPP-ACP either alone or combined with APF could be considered as an alternative prophylactic application in orthodontic practice since it did not compromise bracket bond strength.

KEY WORDS: Shear bond strength; Casein phosphopeptide–amorphous calcium phosphate (CPP-ACP); Topical fluoride

INTRODUCTION

White spot decalcification and caries formation under and around orthodontic bands or brackets are

problems of great concern in orthodontics.¹ Although caries and enamel decalcification can be greatly reduced by maintaining good oral hygiene and using a fluoride-containing dentifrice, use of prophylactic agents are also recommended. Reports suggest that topical fluoride application may reduce or eliminate decalcification during fixed orthodontic treatment.² Several methods of topical fluoride application in orthodontic practice are (1) before etching,^{3,4} (2) during etching,^{5,6} or (3) after etching (before bracket bonding) the enamel.^{7,8}

The mechanism by which fluoride reduces decalcification and caries has also been shown to increase the resistance of enamel to acids.⁹ The fluoride deposits in hydroxyapatite form fluorapatite, which is claimed to affect the bond strength and/or debonded interface.¹⁰ Some studies^{11,12} have reported that topical

^a Research Fellow, Department of Orthodontics, Faculty of Dentistry, Baskent University, Ankara, Turkey.

^b Research Fellow, Department of Pedodontics, Baskent University, Ankara, Turkey.

^c Postgraduate Student, Department of Orthodontics, Faculty of Dentistry, Baskent University, Ankara, Turkey.

^d Professor, Department of Mining and Engineering, Hacettepe University Faculty of Engineering, Ankara, Turkey.

Corresponding author: Dr Sevi Burçak Çehreli, Department of Pedodontics, Baskent University, Ankara, Bahcelievler, Turkey (e-mail: seviburcak@yahoo.com)

Accepted: January 2007. Submitted: December 2006.

© 2008 by The EH Angle Education and Research Foundation, Inc.

application of fluoride can interfere with the bonding mechanism, resulting in reduced bond strength of dental resins. In contrary, other studies¹³⁻¹⁵ demonstrated that the topical application of the fluoride did not adversely affect either the etch pattern on the enamel or the bond strength of composite resin.

Recently, a milk protein derivative, casein phosphopeptide–amorphous calcium phosphate (CPP-ACP) complex, has been introduced for caries prevention and enamel remineralization.¹⁶ The proposed mechanism of action of CPP-ACP is related to its localization at the tooth surface, where it buffers free calcium and phosphate ion activities, maintaining a state of supersaturation with respect to tooth enamel, thereby preventing demineralization and facilitating remineralization.¹⁶ Some recommended professional applications for CPP-ACP complex are white spot prevention/removal in orthodontics, immediately following surgery bleaching, following professional tooth cleaning, after application of topical fluoride, and to provide a topical coating for patients suffering from erosion, caries, and conditions arising from xerostomia.¹⁷

Despite recommendations for its utilization in orthodontics, there are no available data reporting the effects of CPP-ACP on bracket bonding. The aim of this study, therefore, is to evaluate and compare the effects of topical application of CPP-ACP, acidulated phosphate fluoride (APF), and both on shear bond strength of brackets. The null hypothesis is that the shear bond strength of orthodontic brackets is not affected by tested enamel pretreatment methods.

MATERIALS AND METHODS

Forty-eight extracted bovine permanent mandibular incisors were collected from a local slaughterhouse. Immediately after harvesting, the teeth were cleaned of debris and soft tissue remnants and then polished with nonfluoridated pumice and rubber prophylactic cups at low speed for 10 seconds. Tooth selection criteria included absence of any visible irregularity or crack of the enamel surface under 4× magnification and the availability of a macroscopically smooth, flat labial surface suitable for bonding. The teeth were randomly assigned to one of four groups:

- Group 1: Served as control, and no pretreatment was performed on enamel.
- Group 2: Enamel was treated with 1.23% APF (Sultan, Topex, NJ) for 4 minutes and CPP-ACP (Recaldent Tooth Mousse; GC Europe, Leuven, Belgium) for 3 minutes, respectively.
- Group 3: Enamel was treated with CPP-ACP for 3 minutes.
- Group 4: Enamel was treated with 1.23% APF for 4 minutes.

Orthodontic metal brackets (Microarch Standard; GAC International, Bohemia, NY) with a base area of approximately 11.26 mm² were used to bond all teeth.

During and after specimen preparation, the teeth were stored in distilled water at room temperature. To exclude possible differences in bond strength caused by the orthodontic adhesive used, all brackets were bonded with the same material (Transbond XT; 3M Unitek, Monrovia, Calif). Before bonding, each bracket was subjected to a 300-g compressive force for 10 seconds, as described previously by Bishara et al.¹⁸

The excess resin was removed with a small scaler before photopolymerization. A halogen light-curing unit (Hilux; Benlioglu, İstanbul, Turkey) was used for curing the resin, 20 seconds from both the mesial and distal sides. The adequacy of the unit's irradiance was confirmed with a radiometer before photopolymerization.

Specimens were stored in deionized water at 37°C for 24 hours, and then thermal cycling in deionized water was performed at 5°C ± 2°C to 55°C ± 2°C for 1000 cycles. The total period of exposure to both 5°C ± 2°C and 55°C ± 2°C was 10 seconds, with a dwell time of 5 seconds in each bath. The teeth were then kept in distilled water at 37°C for 6 weeks before testing procedures. The water was changed every week. After thermal agitation and water storage, the roots were removed with a low-speed diamond saw under coolant water, and the crowns were embedded in acrylic placed in phenolic rings, with a mounting jig used to align the labial surface of each tooth so that it was perpendicular to the bottom of the mold. Samples were then mounted in the jig attached to the universal testing device (model 4204; Instron, Canton, Mass). For shear testing, the specimens were secured in the lower jaw of the machine so that the bracket base of the sample paralleled the direction of the shear force. The specimens were stressed in an occlusolingival direction with a cross-head speed of 1 mm/min, as in previous studies.^{19,20} The force required to dislodge the bracket was recorded in newtons and converted to megapascals with the following equation: shear force (MPa) = debonding force (N)/[w × l] (mm²), where w = width of the bracket base, l = height of the bracket base, and 1 MPa = 1 N/mm².

After debonding, the teeth and the brackets were examined under a stereomicroscope at 10× magnification for any adhesive remaining, in accordance with the modified adhesive remnant index (ARI).²¹ ARI scores range from 5 to 1, in which 5 = no adherence of composite on enamel, 4 = less than 10% of composite remaining on the enamel, 3 = more than 10% but less than 90% of composite remaining on the enamel, 2 = more than 90% of composite remaining on the enamel, and 1 = all composite remaining on the enamel, with the impression of the bracket base.

Table 1. Descriptive Statistics in Megapascals and Results of Kruskal-Wallis Test Comparing Shear Bond Strength of Four Groups^a

Group				
1 (control)	12	5.37 ± 0.71	5.27	a
2 F+CPP-ACP	12	6.43 ± 0.55	6.57	b
3 CPP-ACP	12	5.98 ± 0.69	5.72	b
4 APF	12	6.46 ± 0.62	6.33	b

^a Identical lettering in the last column indicates values that are not significantly different at $P < .05$.

The Kruskal-Wallis test at $P \leq .05$ was used to determine whether significant differences existed between the shear bond strengths of the groups.²² The χ^2 ($P \leq .05$) test was used to determine significant differences in the ARI scores among the different groups.

RESULTS

The descriptive statistics for the shear bond strengths of the four groups are presented in Table 1. There was a significant difference among the shear bond values of the test groups and the control group ($P = .002$). The bond strengths of the test groups were significantly greater than those of the control group ($P < .05$). There was no statistically significant difference among the groups pretreated with the APF application, CPP-ACP application, or combined application of these agents. All the groups showed a higher percentage of ARI scores of 5, Table 2. The ARI scores for the four groups are listed in Table 3. The χ^2 test results indicated no significant differences among the groups regarding mode of debonding. Enamel detachment was not found in either group. The most frequent debonding occurred in the bracket-resin interface (66% for the control group and 58% for the test group).

DISCUSSION

The question regarding the most appropriate caries prophylactic method in orthodontic practice still merits further research. A recent systematic review reports that the use of topical fluorides in addition to fluoride toothpaste appears to reduce the incidence of decalcification in patients undergoing orthodontic treatment with fixed appliances.²³

Table 2. Intergroup Comparisons and Significance Value (P)

Intergroup Comparison	P
Group 1–2	.000
Group 1–3	.037
Group 1–4	.000
Group 2–3	.084
Group 2–4	.932
Group 3–4	.070

Table 3. Frequency Distribution of Adhesive Remnant Index (ARI) Scores of the Groups

Group	ARI Score				
	1	2	3	4	5
1	—	—	2	2	8
2	—	—	4	1	7
3	—	1	3	1	7
4	—	1	3	1	7

Gwinnett et al found that topically applied fluorides could significantly reduce bond strength by disrupting the formation of enamel tags.^{24,25} These authors have demonstrated the formation of a globular reaction product (possibly CaF) on the etched surface and have recommended thorough rinsing of the enamel after topical fluoride application. In the present study, although every effort was made to remove all the agents from the enamel surface, this was not a great concern since prophylactic agents were applied prior to acid etching of the enamel.

Hirce et al⁷ found that etching enamel for 4 minutes with 50% phosphoric acid containing 2% sodium fluoride significantly weakened the bond strength compared to etching teeth with 50% phosphoric acid alone for 1 minute. The difference in application time is a confounding variable that limits any conclusions about the effect of fluoride on bonding. The decrease in bond strength might be attributed to overetching the enamel and obliterating all enamel tags. Other factors such as variation in the fluoride concentrations used, improvements in the properties of the bonding agents, and/or the bracket retention mechanism could also affect the results.

Contrary to the findings of Hirce et al,⁷ Garcia-Godoy et al¹¹ reported that acid-etching enamel with 60% phosphoric acid containing 0.5% sodium fluoride produced statistically higher shear bond strengths compared with enamel etched with 38% phosphoric acid alone. More recently, Garcia-Godoy²⁶ found no difference in shear bond strength between teeth that were pretreated with APF and teeth that did not receive APF pretreatment.

When the effect of APF pretreatment is considered, the findings of the present study are in accordance with the previous findings of Garcia-Godoy et al.¹¹ Although there was no significant difference among test groups, the fluoride-pretreated enamel demonstrated the higher bond strength that is followed by fluoride + CPP-ACP in the pretreated group. The enhanced bond strength presented herein might be attributed to differences in the study design. Previously reported data were obtained by using extracted human premolars as substrate. Intact human premolars vary in the curvature of their labial surface and represent a complex three-dimensional convex configuration for

bonding with conventional testing methods. Thus, in the present study, bovine teeth were used because bovine enamel has been reported to be a reliable substitute for human enamel in bonding studies, with no statistically significant difference in enamel-bonding value.^{27,28} However, some minor differences among the human enamel and bovine enamel have been reported.

Because bovine enamel and dentin develop more rapidly during tooth formation, bovine enamel has larger crystal grains and more lattice defects than human enamel does.²⁹ This may contribute to a reported lower critical surface tension in bovine enamel than in human enamel.³⁰ These differences might have contributed to the results of the present study.

Reynolds³¹ suggested that a minimum bond strength of 6 to 8 MPa was adequate for most clinical orthodontic needs. These bond strengths are considered able to withstand masticatory and orthodontic forces. In this experiment, all bond strength values achieved were much above this minimal requirement. The results of this study indicated that shear bond strength is favorably affected when the enamel surfaces have been treated with 1.23% APF, CPP-ACP, or their combination.

This study provides preliminary data on the effect of the CPP-ACP on the shear bond strength of brackets. However, one should consider the limitations of in vitro tests when interpreting the results.

CONCLUSIONS

- APF application, CPP-ACP application, and a combined application of these agents may safely be used for caries prophylaxis before bracket bonding when a three-step bonding procedure is used. Further research is indicated to test the effect of these prophylactic applications when self-etch adhesive systems are used.
- The effects of three tested applications on shear bond strength were not significantly different. This finding necessitates conduction of further studies to compare the effectiveness of these methods to choose the best caries prevention method for clinical use in orthodontics.

REFERENCES

1. Artun J, Brobakken BO. Prevalence of carious white spots after orthodontic treatment with multibonded appliances. *Eur J Orthod*. 1986;8:229–234.
2. Shannon IL. Prevention of decalcification in orthodontic patients. *J Clin Orthod*. 1981;15:694–705.
3. Wang WN, Sheen DH. The effect of pretreatment with fluoride on the tensile strength of orthodontic bonding. *Angle Orthod*. 1991;61:31–34.
4. Lehman R, Davidson CL. Loss of surface enamel after acid etching procedures and its relation to fluoride content. *Am J Orthod*. 1981;80:73–82.
5. Bohrer J, Gedalia I. Fluoride concentration in enamel treated with 50% phosphoric acid and NaF with subsequent decalcification in acid gel. *J Dent Res*. 1980;59:1022–1025.
6. Freeman JE, Shannon IL. Addition of stannous fluoride to acid etchant in direct bonding procedures. *Int J Orthod*. 1981;19:13–17.
7. Hirce JD, Sather AH, Chao EY. The effect of topical fluorides, after acid etching of enamel, on the bond strength of directly bonded orthodontic brackets. *Am J Orthod*. 1980;78:444–452.
8. Davidson CL, Bekke-Hoekstra IS. The resistance of superficially sealed enamel to wear and carious attack in vitro. *J Oral Rehabil*. 1980;7:299–305.
9. Mellberg JR, Ripa LW. *Fluoride in Preventive Dentistry: Therapy and Clinical Applications*. Carol Stream, Ill: Quintessence; 1983.
10. Aasende R, Depalola PF, Brudevold F. Effects of daily rinsing and ingestion of fluoride solution upon dental caries and enamel fluoride. *Arch Oral Biol*. 1972;17:1705–1714.
11. Garcia-Godoy F, Hubbard GW, Storey AT. Effect of fluoridated etching gel on enamel morphology and shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 1991;100:163–170.
12. Meng CL, Li CH, Wang WN. Bond strength with APF applied after acid etching. *Am J Orthod Dentofacial Orthop*. 1998;114:510–513.
13. Meng CL, Wang WN, Yeh IS. Fluoridated etching on orthodontic bonding. *Am J Orthod Dentofacial Orthop*. 1997;112:259–262.
14. Bishara SE, Chan D, Abadir EA. The effect on the bonding strength of orthodontic brackets of fluoride application after etching. *Am J Orthod Dentofacial Orthop*. 1989;95:259–260.
15. Wang WN, Sheen DH. The effect of pretreatment with fluoride on the tensile bond strength of orthodontic bonding. *Angle Orthod*. 1991;61:31–34.
16. Reynolds EC. Anticariogenic casein phosphopeptides. *Protein Pept Lett*. 1999;6:295–303.
17. *GC Europe Recaldent Tooth Mousse Product Manual*. GC Corporation, Tokyo, Japan.
18. Bishara SE, Gordan VV, VonWald L, Olson ME. Effect of an acidic primer on shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 1998;114:243–247.
19. Cehreli ZC, Keci D, Kocadereli I. Effect of self-etching primer and adhesive formulations on the shear bond strength of orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 2005;127:573–579.
20. Jobalia SB, Valente RM, de Rijk WG, BeGole EA, Evans CA. Bond strength of visible light-cured glass ionomer orthodontic cement. *Am J Orthod Dentofacial Orthop*. 1997;112:205–208.
21. Olsen ME, Bishara SE, Damon P, Jakobsen JR. Evaluation of Scotchbond multi-purpose and maleic acid as alternative methods of bonding orthodontic brackets. *Am J Orthod Dentofacial Orthop*. 1997;111:498–501.
22. Conover WJ. *Practical Nonparametric Statistics*. New York, NY: John Wiley & Sons; 1980.
23. Chadwick BL, Roy J, Knox J, Treasure ET. The effect of topical fluorides on decalcification in patients with fixed orthodontic appliances: a systematic review. *Am J Orthod Dentofacial Orthop*. 2005;128:601–606.
24. Gwinnett AJ, Buonocore MG, Sheykoleslam Z. Effect of fluoride on etched human and bovine tooth enamel surfaces

- as demonstrated by scanning electron microscopy. *Arch Oral Biol.* 1972;17:271–278.
25. Sheykholeslam Z, Buonocore MG, Gwinnett AJ. Effect of fluorides on the bonding of resins on phosphoric acid-etch bovine enamel. *Arch Oral Biol.* 1972;17:1037–1045.
26. Garcia-Godoy F. Shear bond strength of a resin composite to enamel treated with an APF gel. *Pediatric Dent.* 1993;15:272–274.
27. Nakamichi I, Iwaku M, Fusayama T. Bovine teeth as possible substitutes in the adhesion test. *J Dent Res.* 1983;62:1076–1081.
28. Oesterle LJ, Shellhart WC, Belanger GK. The use of bovine enamel in bonding studies. *Am J Orthod Dentofacial Orthop.* 1998;114:514–519.
29. Moriwaki Y, Kani T, Kozatani T, Tsutsumi S, Shimode N, Yamaga R. The crystallinity change of bovine enamel during maturation. *Jpn J Dent Mat.* 1968;9:78–85.
30. Yu KC, Chang R. Adhesive restorative dental materials II, approaches to achieve adhesion. *National Institutes of Dental Research, US Department of Health, Education and Welfare.* 1966:103–131.
31. Reynolds IR. A review of direct orthodontic bonding. *Br J Orthod.* 1975;2:171–178.